


Identifier: ER-SOP-6.31	Revision: 0	Effective Date: 12/14/01	 <p>ENVIRONMENTAL RESTORATION PROJECT</p> <p>A Department of Energy Environmental Cleanup Program</p>
ER Document Catalog Number: ER2001-0199			
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Environmental Restoration Project
Standard Operating Procedure

for:

Sampling of Subatmospheric Air

Los Alamos

NATIONAL LABORATORY

Los Alamos, New Mexico 87545

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Revision Log

<i>Revision No.</i>	<i>Effective Date</i>	<i>Prepared By</i>	<i>Description of Changes</i>	<i>Affected Pages</i>
R0	12/14/01	Ken Kisiel	Replaces SOPs 6.21, 6.22, and 6.27	All

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Sampling of Subatmospheric Air

1.0 PURPOSE

This Environmental Restoration (ER) Project standard operating procedure (SOP) describes methods for sampling subatmospheric air from vapor ports in monitoring wells and boreholes at Los Alamos National Laboratory (Laboratory). This procedure is applicable for any collection of subsurface air samples for either field screening or fixed analytical laboratory analysis.

2.0 SCOPE

This SOP is a mandatory document and shall be implemented by all ER Project participants when sampling subatmospheric air from boreholes and monitoring wells for the ER Project.

Note: Subcontractors performing work under the ER Project's quality program shall follow this SOP for sampling subatmospheric air or may use their own procedure(s). The subcontractor's procedure must meet the requirements prescribed by the ER Project quality management plan and must be approved by the ER Project's quality program project leader before designated activities begin.

3.0 TRAINING

The **field team leader** (FTL) will ensure that personnel read this procedure before beginning subatmospheric air-sampling activities. This document will familiarize the FTL and field team members with the objectives of each air-sampling activity and with the proper operation of sampling equipment, including the sample train, the Bruel and Kjaer (B&K) photoacoustic multigas analyzer, SUMMA canisters, and the adsorbent columns.

In accordance with QP-2.2, "Personnel Orientation and Training," all **field team members** must document that they have read and understand this procedure.

4.0 DEFINITIONS

Note: A glossary of definitions is located on the ER Project internal homepage <http://erinternal.lanl.gov/WritingGuide.shtml>.

- 4.1 *Absolute pressure*—Pressure measured with reference to absolute zero pressure (as opposed to atmospheric pressure), usually expressed as kPa, mmHg, or psia

- 4.2 Adsorbent columns—These columns contain adsorbents such as silica and are analyzed to determine the concentration of the sorbed constituents. In this procedure, adsorbent columns are used to collect subsurface water vapor to analyze for tritium concentrations in the vapor.
- 4.3 B&K gas analyzer—B&K Model 1302 (B&K) is a portable photoacoustic multigas analyzer. The B&K multigas analyzer detects and quantifies gaseous organic concentration in air.
- 4.4 Carbon dioxide meter—The Vaisala carbon dioxide (CO₂) meter is used to quickly and efficiently determine the CO₂ levels of subsurface air. Monitoring the CO₂ levels can help to determine whether the purge cycle has pulled subsurface air from depth.
- 4.5 Calibration gas—Laboratory-certified organic gas mixes are used for the B&K operational check. Three gas mixes of differing concentrations of three organic gases, trichloroethane (TCA), trichloroethylene (TCE), and tetrachloroethane (PCE), are used.
- 4.6 Gauge pressure—Gauge pressure is a measured pressure that is greater than ambient atmospheric pressure (as opposed to absolute pressure). Zero gauge pressure is equal to ambient atmospheric (barometric) pressure. Gauge pressure is usually expressed as kPa, mmHg, or psig.
- 4.7 Sample train—This instrument provides an interface with the vapor port. The sample train consists of a power source, an inlet line, an outlet line, a valve, a purge pump, and a CO₂ meter. The valve separates the purge pump and the sampling equipment. The inlet line is connected to the vapor port. The outlet line is connected to the sampling equipment.
- 4.8 Site-specific health and safety plan (SSHASP)—A SSHASP is a health and safety plan that is specific to a site or ER-related field activity that has been approved by an ER health and safety representative. This document contains information specific to the activity, including scope of work, relevant history, descriptions of hazards associated with the project site(s), and techniques for exposure mitigation (e.g., personal protective equipment [PPE]) and hazard mitigation.
- 4.9 SUMMA canister—These canisters are specially treated using the SUMMA passivation process. The spherical canisters are evacuated to a negative gauge pressure of approximately 25 inHg. The evacuated canisters provide a passive collection and containment system of laboratory-quality air samples.
- 4.10 Tedlar bag—A Tedlar bag is a gas-sampling container used to collect and contain gases. The Tedlar bag is fitted with an inert valve to introduce the gas sample into the bag. The bag contains calibration gas for the B&K operational check. Each Tedlar Bag used is unique to one gas mix.

- 4.11 Vapor port—A vapor port is tubing that extrudes from ports on top of the borehole. Each port corresponds to a different depth within the borehole.

5.0 BACKGROUND AND PRECAUTIONS

Note: This SOP is to be used in conjunction with an approved SSHASP. Also consult the SSHASP for information on and use of all PPE.

5.1 Background

This section provides a more detailed description of the functionality of each component of the system. Each part of the subatmospheric sampling equipment described below has a vital function.

The sample train is the primary component of the subatmospheric air-sampling system. It provides an interface with the vapor port. Several sampling instruments are necessary for subsurface air sampling. The B&K multigas analyzer quantifies gaseous concentrations of several contaminants in the subsurface air sample. The SUMMA canister captures and contains an air sample for transport to an analytical laboratory. The adsorbent column captures and contains water for tritium analysis by an analytical laboratory.

5.1.1 Sample Train

The sample train interfaces the purge pump and the sampling equipment with the vapor ports. There are two distinct operational functions of the sample train: the purge cycle and the sampling cycle. Before each sampling cycle, vapor ports must be purged of stagnant air in the line. Purging the line ensures that the sample taken is representative of the subsurface air at depth; every sampling activity must include a purge cycle. After purging the line, the sample train is available to interface with the sampling equipment.

5.1.2 B&K Sampling

The B&K multigas analyzer screens air for organic gaseous concentrations. Six factory-installed optical filters within the B&K determine which gases may be analyzed. In the ER Project, the six gases are as follows: TCA, TCE, Freon-11, PCE, CO₂, and water vapor. The instrument quantifies and displays the concentrations of the six gases. Data displayed on the B&K is in units of parts per million (ppm). Before each day's sampling activities the B&K must be tested for operational efficiency by an operational check. The B&K is quick and efficient, making it an ideal instrument for determining extent of contamination in the field.

5.1.3 SUMMA Sampling

The SUMMA canister is an evacuated vessel used for collecting and containing analytical quality air samples. The low pressure inside the canister pulls air inside until a neutral pressure has been achieved. The manufacturer certifies the canisters to be devoid of contaminants, and the inside of the canister is nonreactive in order to preserve the integrity of the air for analysis.

5.1.4 Adsorbent Column Sampling

Tritium levels in soil water vapor can easily be collected and contained for analysis when using silica as an adsorbent. Water vapor is adsorbed onto the silica when subsurface air is pulled through the column. After a sample of subsurface water vapor has been collected, the column is removed from the system and is sealed, providing both a collection and containment vessel. The sealed columns may then be sent to an analytical laboratory for analysis.

5.2 Precautions and Safety Issues

Note: All activities described in this procedure are performed by a **field team member**, unless otherwise specified.

- 5.2.1 Follow properly documented field procedures to ensure that wells and boreholes do not become damaged or contaminated during sampling activities.
- 5.2.2 Waste generated from sampling activities must be handled in accordance with ER-SOP-1.06, "Management of ER Project Wastes."
- 5.2.3 Personnel safety procedures, such as safety practices and site-specific requirements determined by the **site safety officer** (SSO) and the SSHASP, must be observed to prevent exposure to hazardous materials and physical hazards.
- 5.2.4 This procedure requires the use of compressed-gas cylinders, pumps, and field-screening instruments. All equipment and materials must be handled in a safe manner consistent with the limitations stated by the manufacturer. Carefully read all warning labels associated with the equipment. Obtain a material safety data sheet (MSDS) for all compressed gases and reagents from the SSO or manufacturer. The **FTL** shall ensure all field team members have reviewed the MSDS of each gas before starting sampling operations.

- 5.2.5 Vapor ports extend from the borehole cover and are connected to lines that descend through boreholes. Care should be taken when handling the vapor ports. Because of the harsh conditions in the field, the plastic tubing of the ports may degrade over time. The depth tags on the vapor port may become unreadable. Plugs may be lost. Document any unusual conditions of the vapor ports in the field logbook.
- 5.2.6 Special care should be taken during the installation of the adsorbent columns into the sample train. The adsorbent columns should be handled carefully to minimize exposure to ambient air because of contamination that might be present. The adsorbent columns must be properly oriented in the train so that the air stream flows into the column through the designated inlet line.
- 5.2.7 Radon contamination is present on some of the vapor ports. Invariably radon will collect on the vapor ports because the plastic has a slightly negative static charge. When connecting the ports to the sample train, the field team member may receive a radon dose, particularly from vapor ports at ground level. The human body has a slight positive charge, and because of contact with the vapor ports, the hands may attract minute amounts of radon. Simply clapping the hands to relieve any built-up static charge may alleviate this problem.

6.0 RESPONSIBLE PERSONNEL

The following personnel are responsible for activities identified in this procedure.

- 6.1 Field Team Leader (FTL)
- 6.2 Field team member
- 6.3 SSO

7.0 EQUIPMENT

This section describes the equipment and discussed its advantages, disadvantages, and limitations. Refer to the background section for a description of the operational functionality of each field-sampling instrument.

7.1 Sample Train

The sample train consists of a power source, an inlet line, an outlet line, a valve, a purge pump, and a CO₂ meter. The power source is a four-outlet surge protector at 120 V AC that powers the purge pump, the CO₂ meter, and the B&K. The inlet line interfaces with the vapor ports by plugging into and onto the ports. The outlet line sends air to the sampling equipment. The

valve switches the inlet line between the purge pump and the outlet line. The purge pump pulls subsurface air into the CO₂ meter. The meter displays the CO₂ levels of the subsurface air. When CO₂ levels stabilize, the subsurface air in line is sampled. Most sampling devices can be utilized for subsurface air sampling using the sample train.

The sample train is a delicate device. The tubing can kink and allow areas for contaminants to gather and contaminate sampling. The tubing must also remain free of dirt and debris that may foul or plug the inlet/outlet lines. Once sampling is complete, the lines must be purged for 10 to 15 minutes to remove contaminants within the system.

To ensure valid results, nonmetal tubing must be replaced quarterly. Replacement of the tubing must be documented in the field logbook. If tubing was not been replaced after the previous sampling event, it must be replaced and the replacement documented in the field logbook before quarterly sampling activities begin.

7.2 B&K Photoacoustic Multigas Analyzer

The B&K analyzer quantifies gaseous concentration of six constituent gases in air samples. The analysis cycle lasts approximately two minutes and consists of an internal purge, a sampling event, an analysis event, and finally a display. The internal purge expels all the previous sample air within the analysis chamber. The sampling event draws a new air sample into the analysis chamber. The analysis event is the photoacoustic interaction between the air and the infrared light within the analysis chamber. The analysis chamber is a vessel that houses an optical filter and a microphone. The filter allows infrared light to pass into the air sample. The microphones listen for the photoacoustic interaction. The internal computer quantifies the analysis, and then the instrument displays the results.

The B&K must be tested for functionality by an operational check before field-sampling events begin.

The B&K should avoid the following conditions or events that may hinder performance.

- The B&K is temperature sensitive and must be protected from thermal trauma.
- The microphones make the B&K shock sensitive; hence, the B&K must avoid intense physical trauma.
- The B&K tubing must remain free of debris and dirt that can foul the internal pumps and the internal air filters.

7.3 SUMMA Canisters

The SUMMA sample canister is a stainless steel canister evacuated to a negative pressure of approximately 25 inHg. The passivation process of the stainless steel canister ensures that it will not react with constituents in the sample. The low pressure of the canister also eliminates the need for a pump to draw the sample. The stainless steel design and evacuation provide a simple, efficient method for providing an analytical-quality subsurface air sample.

The connection of the SUMMA canister to the sample train must not be compromised. A pressure valve and vacuum gauge help ensure there are no leaks in the system. The pressure valve and vacuum gauge also regulate the rate and duration of air collection into the canister. The vacuum gauge aids in determining if leaks are present in the pressure valve, a well port is blocked, or if the SUMMA is full.

To ensure sample quality, SUMMA canisters must be certified by the contract laboratory as clean and leak free. Certified clean canisters must be obtained through the Laboratory's Sample Management Office (SMO) from the contract laboratory where the samples will be analyzed.

7.4 Adsorbent Columns

The adsorbent columns are cylinder containers filled with silica and open at each end. A pump pulls air through the silica. Water vapor from the subatmospheric air adsorbs onto the silica surface. After an appropriate amount of water vapor has been collected, the column is sealed at each end. The column provides both collection and containment vessel.

The silica, once placed in the column, must not be exposed to ambient air. Excessive exposure to ambient air may allow ambient water vapor to collect thus spoiling the adsorbent column sampling. The ends of the columns must immediately be sealed after sample collection is complete.

The mass of the column is vital for analysis. The mass of the column must be measured before field activities begin or immediately before sampling. Take note of the mass of the column plugs as well.

7.5 Boreholes and Vapor Ports

Boreholes are deep wells that have six to eight vapor ports. Borehole identification numbers are typically stamped or displayed on the lid. The ports protrude from the top of the borehole cap and are labeled to identify the depth of the port. Each port has an extruding section of plastic tubing for connection with the sample train.

7.6 Hand Tools

The following tools are necessary, but other tools may also be useful.

- Set of crescent wrenches
- Vise grips
- One-quarter-inch Teflon tubing
- Teflon tape
- One-quarter-inch Swagelok fittings (ferrules, collets, bolts, connectors)
- Pipe wrench

8.0 PROCEDURE

Note: ER Project personnel may produce paper copies of this procedure printed from the controlled-document electronic file located at http://erinternal.lanl.gov/home_links/Library_proc.shtml. However, it is each person's responsibility to ensure that they trained and are using the current version of this procedure. The author may be contacted if text is unclear. The document control coordinator may be contacted if the author cannot be located.

Note: Deviations from SOPs are made in accordance with QP-4.2, "Standard Operating Procedure Development," and documented in accordance with QP-5.7, "Notebook Documentation for Environmental Restoration Technical Activities."

Note: All activities described in this procedure are performed by a **field team member**, unless otherwise specified.

Note: All field logbook entries are documented in accordance with QP-5.7, "Notebook Documentation for Environmental Restoration Technical Activities."

8.1 Presampling Activities

- 8.1.1 Identify appropriate sampling techniques to be used (the B&K samples to determine the extent of contamination of chlorinated organic vapor in the subsurface air, the SUMMA canisters to collect laboratory-quality air samples and contain them for shipping, or the adsorbent columns to sample subsurface water vapor in subsurface air).
- 8.1.2 Inspect all tubing, fittings, and valves on the sample train. Inspect Swagelok fittings for degradation. Tighten, as necessary, all fittings and valves that make up the assembly. Ensure that the power supply is functional.
- 8.1.3 Perform B&K operational check, if needed.

Note: The purpose of the operational check is to introduce a laboratory-certified organic gas mixture to the B&K to check the unit for functionality. Three mixes of the calibration gas are sampled by the B&K. If the B&K reports a concentration equal to or better than 80% of the laboratory-certified concentration, then the B&K is considered functional. If the B&K operational check does not quantify the results of the analysis within 80% of the laboratory-certified concentrations, several actions may be taken to improve performance.

- Changing the setup parameter. Refer to B&K 1305 operational manual for instructions.
- Inspecting the Tedlar bags. Bags may degrade and fail over time and allow leaks.
- Calling the manufacturer (California Instruments) at (714) 974-5560.

Note: Before operating the B&K unit, read the operational instruction manual of the B&K 1305 unit. Setup conditions of the B&K may need to be changed from time to time. Refer to the B&K 1305 operational manual for definitions of error messages.

8.1.3.1 Ensure that the following equipment is available:

B&K unit

CGA 590 bolt compressed gas regulator

three Tedlar bags

a large adjustable wrench

a length of one-quarter-inch Teflon tubing

one-quarter-inch Swagelok fittings (bolts, ferrules, collets)

calibration gas (Table 1)

Table 1
Examples of Three Point Calibration Gases Used for
Quarterly Subatmospheric Air Sampling at Technical Area 54

Organic Gases	Low Concentration (ppm)	Medium Concentration (ppm)	High Concentration (ppm)
TCA	50	100	480
TCE	10	50	200
PCE	5	10	50

- 8.1.3.2 Inspect the on/off switch, the functional buttons, power cord, the inlet line and the outlet line. Ensure the B&K has a power source that is functional.
- 8.1.3.3 The first step in the operational check is to fill the Tedlar bags.

Note: The Tedlar bags are to be filled with gases contained in pressurized tanks. Great care should be taken when handling the pressurized tanks.

 - 8.1.3.3.1 Identify the concentration of the calibration gas. Confirm that the calibration gas concentration and the Tedlar Bag calibration gas concentration label agree. Each bag should only be used for a specific gas mix.
 - 8.1.3.3.2 Ensure that the regulator valve is closed. Then connect the regulator to the calibration gas bottle, and connect the Tedlar bag to the regulator.
 - 8.1.3.3.3 Open the valve on the Tedlar bag and the valve on the bottle.
 - 8.1.3.3.4 Slowly open the regulator valve and fill the Tedlar bag. Close the regulator valve, the Tedlar bag valve, and then the bottle valve.
 - 8.1.3.3.5 Remove the Tedlar bag from the regulator. Open and close the regulator valve to release any gas within the regulator.
 - 8.1.3.3.6 Remove the regulator from the gas bottle.
 - 8.1.3.3.7 Repeat the procedure (Steps 8.1.3.3.1 through 8.1.3.3.6) by filling the other Tedlar bags with the other calibration gas mixtures.
- 8.1.3.4 The second step in the operational check is to perform the B&K sampling.
 - 8.1.3.4.1 Turn on the B&K.
 - 8.1.3.4.2 Connect a Tedlar bag to the B&K inlet line. Open the Tedlar bag valve.
 - 8.1.3.4.3 Begin continuous monitoring of the B&K. Refer to the B&K manual for B&K operation instructions.

- 8.1.3.4.4 Allow the B&K to take several samples of gas from the Tedlar bag.
- 8.1.3.4.5 Observe the B&K gas concentration display. The goal is to achieve concentration values equal to or greater than 80% of the laboratory-certified concentrations of the calibration gas mix. Document the operational check results into the field logbook.
- 8.1.3.4.6 Close the Tedlar bag valve, and remove it from the inlet line.
- 8.1.3.4.7 Repeat steps 8.1.3.4.2 through 8.1.3.4.8 using the next calibration gas mix.
- 8.1.3.4.8 Document the six gas concentration values of each calibration gas mix (there are three) in the field logbook under the B&K operational check.
- 8.1.4 Document the following presampling activities into the field logbook: sample train inspection, calibration, port conditions, tubing problems/solutions, and B&K operational check.
- 8.1.5 Mobilize to site.
- 8.2 Sampling Activities
 - 8.2.1 Confirm borehole number and location. Document commencement of sampling activities in field logbook. Identify and correlate borehole number with field logbook borehole number.
 - 8.2.2 Inspect the vapor port. Document any abnormal conditions of the vapor port in the field logbook.
 - 8.2.3 Purge the sample train for approximately 10 to 15 minutes with the purge pump to remove all stagnant air within the tubing and valves.
 - 8.2.4 Connect the sample train inlet line to the vapor port.
 - 8.2.5 Begin one of the specific sampling activities below.
 - 8.2.6 B&K Sampling
 - 8.2.6.1 Disconnect the B&K inlet line from the sample train. Ensure that the B&K is in *Continuous Monitoring* mode. Press *Standby* button on B&K control display. This starts the sampling cycle.

- 8.2.6.2 Allow the B&K to take three ambient air readings, and record gas concentrations for each in the field logbook.
- 8.2.6.3 Press *Standby* button. This stops the sampling cycle.
- 8.2.6.4 Connect the B&K inlet line to the B&K outlet of the sample train.
- 8.2.6.5 Ensure that the sample train valve is turned to *Purge*.
- 8.2.6.6 Ensure that the CO₂ meter inlet line is connected to the purge pump outlet line and is operating. Activate the purge pump and purge vapor port to depth. Observe the CO₂ measurement carefully while purging the line. When the CO₂ level stabilizes, read and record the measurement into the field logbook.
- 8.2.6.7 Deactivate the pump and quickly turn the sample train valve from *Purge* to *B&K*.
- 8.2.6.8 Press the *Standby* button on the B&K control panel to start the sampling cycle.
- 8.2.6.9 Record the measurement of the B&K analysis and the current date/time in the field logbook.
- 8.2.6.10 Press *Standby* button to stop the sampling.
- 8.2.6.11 Disconnect sample train inlet line from vapor port.
- 8.2.7 SUMMA Sampling
 - Note:** Ensure that steps 8.2.1 through 8.2.5 have been completed.
 - 8.2.7.1 Activate purge pump and purge vapor port to depth. Observe CO₂ measurement carefully while purging the line. When the CO₂ level stabilizes, read and record measurement into field logbook.
 - 8.2.7.2 Disconnect the sample train from the vapor port.
 - 8.2.7.3 Connect pressure valve with the vacuum gauge to the vapor port. Ensure all valves are closed.
 - 8.2.7.4 Attach SUMMA canister to pressure valve.
 - 8.2.7.5 Open the valve on the SUMMA canister, and check the vacuum gauge for proper vacuum.
 - 8.2.7.6 Open the pressure valve. The SUMMA canister will draw a air sample because of the vacuum in the canister.
 - 8.2.7.7 Close the valve on the canister when the gauge indicates that the pressure in the canister and atmospheric pressure

have equilibrated. Complete the identification tag of the canister.

8.2.7.8 Document SUMMA sampling in the field logbook, in the sample collection log, and on the chain of custody forms.

8.2.7.9 Disconnect pressure valve from vapor port.

8.2.7.10 Store the canister in the shipping container and ship to the Laboratory SMO, according to ER SOP 1.03, "Handling, Packaging, and Shipping of Samples."

8.2.8 Adsorbent Column Sampling

Note: Ensure that steps 8.2.1 through 8.2.5 have been completed.

8.2.8.1 Measure the mass of the adsorbent columns before field activities, and document in the field logbook.

8.2.8.2 Activate purge pump and purge vapor port to depth. Observe CO₂ measurement carefully while purging the line. When the CO₂ level stabilizes, read and record measurement into field logbook.

8.2.8.3 Connect the adsorbent column to the exhaust of the sample train.

8.2.8.4 Activate pump to pull air through the adsorbent column for a specified time. The **FTL** should designate the time needed for adequate sample recovery.

8.2.8.5 Quickly remove column, and seal the ends.

8.2.8.6 Document adsorbent column sample in the field logbook, in the sample collection log, and on the chain of custody.

8.2.8.7 Submit samples to the Laboratory SMO, according to ER SOP 1.03, "Handling, Packaging, and Shipping of Samples."

8.3 Postsampling Activities

Dispose of any tubing that is visibly damaged or contaminated.

8.4 Lessons Learned

Note: During the performance of work, ER Project personnel shall identify, document, and submit lessons learned, as appropriate, in accordance with QP-3.2, "Lessons Learned," located at http://erinternal.lanl.gov/home_links/Library_proc.shtml.

- 8.4.1 Purge indicator gas concentrations (i.e., CO₂) should remain constant if the sample stream is free of leaks and a proper purge is achieved.
- 8.4.2 Ensure quality of the sample by eliminating any leaks within the system. Whenever connecting two or more gas lines, confirm that the connection is free of visible and audible leaks. Whenever data quality is questioned or possibly compromised, inspect all connections for leaks.

9.0 REFERENCES

ER Project personnel using this procedure should become familiar with the contents of the following documents to properly implement this SOP.

ER Project Quality Management Plan located at
http://erinternal.lanl.gov/home_links/Library_proc.shtml

The following documents are cited within this procedure.

QP-2.2, Personnel Orientation and Training

QP 3.2, Lessons Learned

QP-4.2, Standard Operating Procedure Development

QP-4.4, Record Transmittal to the Records Processing Facility) to the Records Processing Facility

QP-5.7, Notebook Documentation for Environmental Restoration Technical Activities

ER SOP 1.03, Handling, Packaging and Shipping of Samples

ER-SOP-1.06, Management of ER Project Wastes

10.0 RECORDS

The **FTL** is responsible for submitting the following records (processed in accordance with QP-4.4, Record Transmittal to the Records Processing Facility) to the Records Processing Facility.

10.1 Completed chain-of-custody/request for analysis form

10.2 Field logbook

10.3 Sample collection log

11.0 ATTACHMENTS

None